

Claims:

1. A method for preparing microparticles comprising an aqueous phase comprising an aqueous solution of a water-soluble oil or gas field production chemical or an aqueous dispersion of a water-dispersible oil or gas field production chemical encapsulated in a continuous polymeric phase comprising a degradable polymer, which
5 method comprises:
 - a) preparing a water in oil in water double emulsion or water in oil in oil double emulsion having an internal aqueous phase comprising an aqueous solution of a water-soluble oil or gas field production chemical or an aqueous dispersion of a water-dispersible oil or gas field production chemical and an internal oil phase comprising a
10 solution of a degradable polymer dissolved in a volatile organic solvent wherein the internal aqueous phase is dispersed in the internal oil phase of the double emulsion in the form of droplets having a mean diameter of less than 10 microns; and
 - b) at least in part evaporating the volatile organic solvent from the double emulsion such that the degradable polymer accumulates around the droplets of the internal
15 aqueous phase and hardens to form microparticles comprising a continuous polymeric phase encapsulating the internal aqueous phase having a mean particle diameter of less than 10 microns.
2. A method as claimed in Claim 1 wherein the oil or gas field production chemical is dissolved or dispersed in the in the internal aqueous phase of the microparticles in an
20 amount in the range of from 1 to 50 percent by weight, preferably 5 to 30 percent by weight.
3. A method as claimed in Claims 1 or 2 wherein the oil or gas field production

chemical is selected from the group consisting of water-soluble or water-dispersible (i) scale inhibitors, (ii) corrosion inhibitors, (iii) hydrogen sulphide scavengers and (iv) hydrate inhibitors.

4. A method as claimed in any one of the preceding claims wherein the internal aqueous phase of the double emulsion prepared in step a) is dispersed in the internal oil phase in the form of droplets having a mean diameter of less than 1 micron and the microparticles formed in step b) have a mean diameter of less than 1 micron.

5. A method as claimed in any one of the preceding claims wherein the microparticles are microcapsules or microspheres.

10 6. A method as claimed in any one of the preceding claims wherein the volatile organic solvent is evaporated in step b) by stirring the double emulsion at a temperature at least 10°C below the boiling point of the volatile organic solvent.

7. A method as claimed in Claim 6 wherein the double emulsion is subjected to sonication during step b).

15 8. A method as claimed in any one of the preceding claims wherein the volatile organic solvent has a boiling point of less than 80°C.

9. A method as claimed in Claim 8 wherein the volatile organic solvent is selected from the group consisting of alcohols, acetone, ethers, ketones, halogenated hydrocarbons, ethyl acetate, alkyl sulfoxides, dimethylamine, dimethyl formamide, and
20 N-methyl-2-pyrrolidone.

10. A method as claimed in any one of the preceding claims wherein the degradable polymer is dissolved in the internal oil phase of the double emulsion in an amount in the range 1% to 50% by weight, preferably 2% to 15% by weight.

11. A method as claimed in any one of the preceding claims wherein the microparticles are collected by filtration, centrifugation, dialysis, evaporation of the dispersing medium, spray drying, or freeze drying.

12. A method as claimed in any one of the preceding claims wherein the microparticles are prepared by:

a) mixing an aqueous phase comprising an aqueous solution of a water-soluble oil or gas field production chemical or an aqueous dispersion of a water-dispersible oil or gas field production chemical with a primary oil phase comprising a solution of a polymer
30 in a volatile organic solvent in the presence of a water-soluble surfactant to form a water

in primary oil emulsion having the aqueous phase dispersed in the primary oil phase in the form of droplets having a mean diameter of less than 10 microns;

b) mixing the water in primary oil emulsion with a secondary oil phase in the presence of an oil-soluble surfactant to form a water in primary oil in secondary oil

5 double emulsion; and

c) at least in part evaporating the volatile organic solvent from the water in primary oil in secondary oil double emulsion such that the polymer accumulates around the droplets of the aqueous phase and hardens to form microparticles comprising a continuous polymeric phase encapsulating the aqueous phase having a mean particle

10 diameter of less than 10 microns.

13. A method as claimed in Claim 12 wherein the water in primary oil emulsion is dispersed in the secondary oil phase of the water in primary oil in secondary oil double emulsion in the form of droplets having a mean diameter of less than 50 microns, preferably, less than 25 microns, more preferably less than 10 microns.

15 14. A method as claimed in any one of Claims 1 to 11 wherein the microparticles are prepared by:

a) mixing a primary aqueous phase comprising an aqueous solution of an water-soluble oil or gas field production chemical or an aqueous dispersion of a water-dispersible oil or gas field production chemical with an oil phase comprising a solution

20 of a polymer dissolved in a volatile organic solvent in the presence of a first water-soluble surfactant to form a water in oil emulsion having the primary aqueous phase dispersed in the oil phase in the form of droplets having a mean diameter of less than 10 microns;

b) mixing the water in oil emulsion with a secondary aqueous phase in the presence

25 of a second water-soluble surfactant to form a water in oil in water double emulsion; and

c) at least in part evaporating the volatile organic solvent from the water in oil in water double emulsion such that the polymer accumulates around the droplets of the primary aqueous phase and hardens to form microparticles comprising a continuous

30 polymeric phase encapsulating the aqueous phase having a mean particle diameter of less than 10 microns.

15. A method as claimed in Claim 14 wherein the water in oil emulsion is dispersed in

the secondary aqueous phase of the water in oil in water double emulsion in the form of droplets having a mean diameter of less than 50 microns, preferably, less than 25 microns, more preferably less than 10 microns.

16. A method as claimed in any one of Claims 12 to 15 wherein the aqueous phase is dispersed in the oil phase of the emulsion of step a) in the form of droplets having a mean diameter of less than 5 microns, preferably, less than 1 micron.

17. A method as claimed in any one of Claims 12 to 16 wherein the water-soluble surfactant employed in step a) has a hydrophilic/lipophilic balance (HLB) value of 8 to 19.

18. A method as claimed in any one of Claims 12 to 17 wherein the internal aqueous phase of the emulsion formed in step (a) is from 10 to 70 percent of the total volume of the emulsion.

19. A method as claimed in any one of Claims 12 to 13 and 16 to 18 wherein the oil-soluble surfactant employed in step b) has an HLB value of less than 8.

20. A method as claimed in any one of Claims 12 to 13 and 16 to 19 wherein the secondary oil phase employed in step b) is selected from the group consisting of alkanes having from 10 to 16 carbon atoms, vegetable oils, and synthetic oils.

21. A method as claimed in Claims 14 to 18 wherein the second water-soluble surfactant of step b) has an HLB value of 8 to 19 and is the same or different to the first water-soluble surfactant of step a).

22. A method of introducing an oil or gas field production chemical into a subterranean formation comprising:

(a) injecting a suspension comprising microparticles suspended in a liquid medium into a formation through an injection well wherein the microparticles comprise an aqueous phase comprising an aqueous solution of a water-soluble oil or gas field production chemical or an aqueous dispersion of water-dispersible oil or gas field production chemical encapsulated in a continuous polymeric phase having a mean diameter of less than 10 microns, preferably less than 5 microns, more preferably less than 1 micron, and the polymer forming the continuous polymer phase is degradable under the conditions encountered in the formation;

(b) allowing the suspension to percolate through the formation towards a production well; and

(c) controllably releasing the aqueous solution of the water-soluble oil or gas field production chemical or the aqueous dispersion of the water-dispersible oil or gas field production chemical from the microparticles into the formation and/or the production well through degradation of the polymer forming the continuous polymeric phase.

5 23. A method as claimed in Claim 22 wherein the microparticles are obtainable by the method of any one of claims 1 to 21.

24. A method as claimed in Claims 22 or 23 wherein the microparticles start to release the aqueous solution or aqueous dispersion of the oil or gas field production chemical at a threshold temperature in the range 50 to 150°C.

10 25. A method as claimed in any one of Claims 22 to 24 wherein the suspension propagates through the formation at a rate of 15 to 100 feet per day.

26. A method of introducing an oil or gas field production chemical into a formation comprising the steps of:

a) injecting a suspension comprising microparticles suspended in a liquid medium
15 into a formation through a production well wherein the microparticles are obtainable by the method of any one of Claims 1 to 21; and

b) shutting in the production well for a period of 2-50 hours before returning the well to production.

20 27. A suspension comprising microparticles suspended in a liquid medium wherein the microparticles are obtainable by the method of any one of Claims 1 to 21.

28. A suspension as claimed in Claim 27 wherein the liquid medium is an oil, an organic solvent or water.

29. A suspension as claimed in Claims 27 or 28 wherein the microparticles are dispersed in the liquid medium in an amount of from 20 to 50% by weight.

25 30. A microparticle obtainable by the method of any one of Claims 1 to 21.

31. A microcapsule comprising a well-defined core of an aqueous solution of a water-soluble oil or gas field production chemical or an aqueous dispersion of water-dispersible oil or gas field production chemical, and a well-defined wall comprising a degradable polymer wherein the microcapsule has a diameter of less than 10 microns,
30 preferably less than 5 microns, most preferably less than 1 micron.